solution was dried, concentrated, and distilled to give cyclohexanone-2,2-d_2. Reduction as described above yielded cyclohexanol-2,2-d_2 (41% yield). The low yield was probably the result of too vigorous exchange conditions.

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Reactions of Phosphorus Compounds. 35. Reaction of 4-Salicyloxybutyltriphenylphosphonium Bromide with Alcoholic Alkoxide

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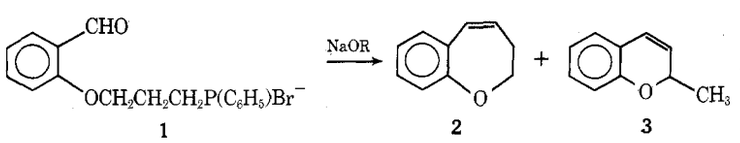
The reaction of 4-salicyloxybutyltriphenylphosphonium bromide (5) with alcoholic alkoxide gave 3,4-dihydro-2*H*-1-benzoxocin (6), 2-ethyl-2*H*-1-benzopyran (7), and 4-(*o*-α,α-diethoxymethylphenoxy)butyldiphenylphosphine oxide (8a). Mechanisms are proposed for the formation of 7 and 8.

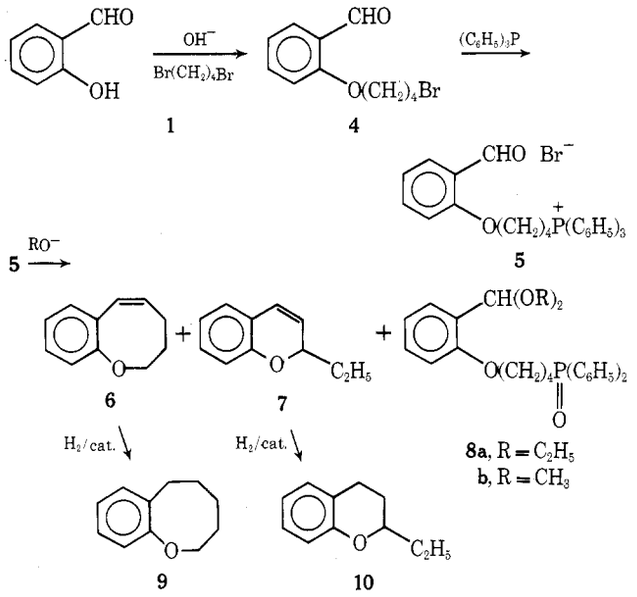
In previous papers[1] we have shown that 3-salicyloxypropyltriphenylphosphonium bromide (1) with base gives either 2,3-dihydro-1-benzoxepin (2) or 2-methyl-2*H*-1-benzopyran (3), depending on the nature of the solvent, and we have proposed a mechanism for the formation of 3 from 1.[1c]



1 2 3

In the present work we wish to report the reactions of 4-salicyloxybutyltriphenylphosphonium bromide (5), under the influence of base, to give the expected 3,4-dihydro-2*H*-1-benzoxocin (6), 2-ethyl-2*H*-1-benzopyran (7), and the unexpected 4-(*o*-α,α-diethoxymethylphenoxy)butyldiphenylphosphine oxide (8a) [or 4-(*o*-α,α-dimethoxymethylphenoxy)butyldiphenylphosphine oxide (8b)].

The reaction of salicylaldehyde with 1,4-dibromobutane in aqueous NaOH gave a 62% yield of 4-salicyloxybutyl



bromide (4). The quaternization of triphenylphosphine by halide 4 gave the phosphonium salt 5 (70%).

Reaction of the salt 5 in refluxing ethanolic sodium ethoxide gave a 19% yield of a mixture of 3,4-dihydro-2*H*-1-benzoxocin (6) and 2-ethyl-2*H*-1-benzopyran (7), in a ratio of 64:36, respectively, and a 32% yield of 4-(*o*-α,α-diethoxymethylphenoxy)butyldiphenylphosphine oxide (8a). The ratio, and yields, of 6 and 7 obtained, using different solvents, are tabulated in Table I. It may be noted, in contrast, that on treating salt 1 in pure MeOH and DMF as solvents the benzopyran 3 and benzoxepin 2, respectively, were obtained (exclusively and in high yield), and no acetal phosphine oxide, comparable to 8, was observed.[1]

Table I
Yields of 6, 7, and 8 from Salt 5

Solvent	Benzoxocin 6 and benzopyran 7 — Temp, °C	Yield, %	Ratio of 6:7[b]	Yield of 8, %
MeOH	Reflux	1–3	63:37	48 (8b)
EtOH	Reflux	19	64:36	33 (8a)
EtOD	Reflux	13	94[c]:6	
DMF[a]	65	19	95:5	
DMF	127	31	98:2	

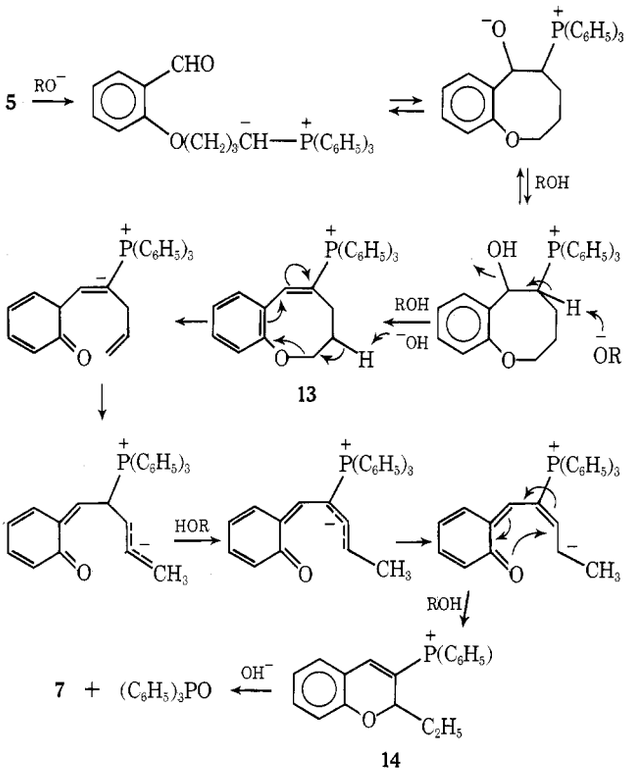
[a] *N*,*N*-Dimethylformamide. [b] By glc. [c] 5-Deuterio substituted 6.

The structure of 3,4-dihydro-2*H*-1-benzoxocin (6) was supported by its reduction to the saturated heterocycle 9, and by examination of the physical data for 6 and 9.

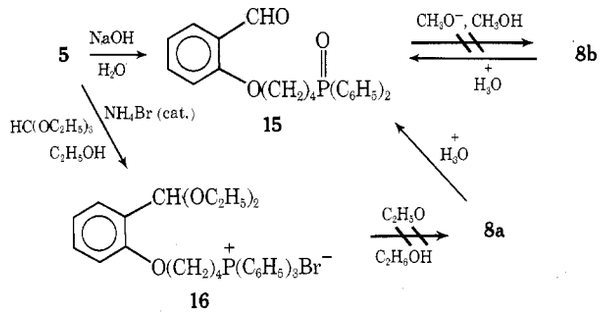
The structure of 2-ethyl-2*H*-1-benzopyran (7) was proven by comparison with an authentic sample prepared in these laboratories[2] from crotyltriphenylphosphonium chloride (11) and sodium salicyloxide (12). Further support for structure 7 was obtained by its reduction to 2-ethylbenzo[*b*]dihydropyran (10), a previously known compound.[3]

We propose that the mechanism for the formation of 2-ethyl-2*H*-1-benzopyran (7) is essentially parallel to that proposed for the formation of 2-methyl-2*H*-1-benzopyran (3) from 3-salicyloxypropyltriphenylphosphonium bromide (1).

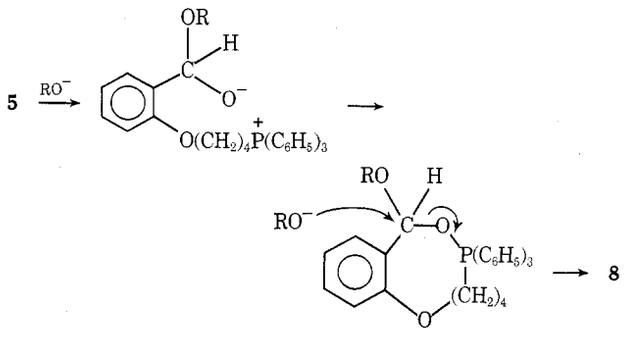
Attempts to isolate intermediate vinylphosphonium salts (13 or 14) under conditions employing catalytic amounts of base, as was accomplished successfully in the series starting with salt 1,[1c] were not successful and yielded only starting material 5. Thus the mechanism rests solely on previous work.[1c]



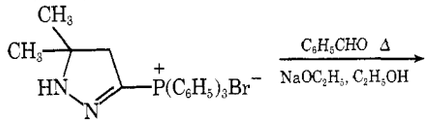
Treatment of 4-(*o*-α,α-diethoxymethylphenoxy)butyldiphenylphosphine oxide (8a), or the dimethyl acetal 8b, with dilute hydrochloric acid gave an essentially quantitative yield of 4-(*o*-methylphenoxy)butyldiphenylphosphine oxide (15). An authentic sample of compound 15 was obtained from 4-salicyloxybutyltriphenylphosphonium bromide (5) by aqueous alkaline hydrolysis.



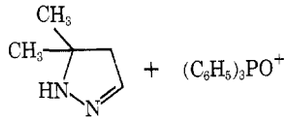
The phosphine oxide 15 did not give the dimethyl acetal 8b on treatment with methanolic sodium methoxide. It was also observed that 4-(*o*-α,α-diethoxymethylphenoxy)butyltriphenylphosphonium bromide (16), prepared from 5 by its reaction with ethyl orthoformate and a catalytic amount of ammonium bromide, did not give the diethyl acetal 8a on treatment with ethanolic sodium ethoxide. These data suggest the mechanism shown below.



A similar acetal formation was observed[4] on allowing 5,5-dimethyl-2-pyrazolin-3-yltriphenylphosphonium bromide (17) to react with benzaldehyde in alcoholic sodium ethoxide. The search for a suitable simple phosphonium salt which may be used to produce acetals and ketals under basic conditions is underway.



17



$C_6H_5CH(OC_2H_5)_2$ (80%)

Experimental Section[5]

4-(*o*-Formylphenoxy)butyl Bromide (4). An aqueous solution of NaOH (65.9 g, 1.6 mol) was added over a period of 2 hr to a rapidly stirred refluxing mixture of 454 g (2.1 mol) of 1,4-dibromobutane and 200 g (1.6 mol) of salicylaldehyde in 950 ml of water. After the reaction had continued for 48 hr the mixture was cooled, and the organic phase was separated and added to 500 ml of chloroform. The organic solution was washed with 4 × 300 ml of a 10% NaOH (aqueous) solution, 300 ml of 10% H_2SO_4 (aqueous), and 2 × 300 ml of H_2O. After the organic phase was dried ($MgSO_4$) and the residue was concentrated, distillation gave 220 g (62%) of **4**, bp 140–180° (2 mm), n^{25}D 1.5645.

An analytical sample, from a similar reaction, had bp 140° (0.2 mm); n^{25}D 1.5617; nmr (CCl_4) δ 1.8–2.4 (m, 4, $C_{2,3}$ methylenes), 3.48 (t, 2, C_1 methylene), 4.06 (t, 2, C_4 methylene), 6.8–7.9 (m, 4, C_6H_4), 10.5 ppm (s, 1, CHO).

Anal. Calcd for $C_{11}H_{13}BrO_2$: C, 51.39; H, 5.06. Found: C, 51.18; H, 5.29.

4-(*o*-Formylphenoxy)butyltriphenylphosphonium Bromide (5). Triphenylphosphine (0.98 mol) and an equimolar amount of **4** were allowed to react for 3 days in refluxing ethyl acetate (1 l.). The mixture was filtered hot and the residue was washed with 2 × 150 ml of hot ethyl acetate and 2 × 150 ml of anhydrous ether to give 353 g (70%) of vacuum oven (80°, 5 mm) dried salt, **5**: mp 157–158°; nmr ($DCCl_3$) δ 1.7–2.5 (m, 4, $C_{2,3}$ methylene), 3.7–4.4 (m, 4, $C_{1,4}$ methylene), 6.77–8.15 (m, 19, aromatic), 10.3 ppm (s, 1, CHO).

Anal. Calcd for $C_{29}H_{28}BrO_2P$: C, 67.17; H, 5.41; P, 5.96. Found: C, 66.92; H, 5.62; P, 601.

Reaction of 4-(*o*-Formylphenoxy)butyltriphenylphosphonium Bromide (5) with Sodium Ethoxide in Ethanol. The salt **5** (42 g, 0.08 mol), dissolved in 250 ml of anhydrous ethanol, was added, over a period of 3 hr, to a refluxing, stirred solution of 0.16 mol of sodium ethoxide in anhydrous ethanol. After the reaction was allowed to proceed for 48 hr the solution was concentrated to 50 ml and added to 2 l. of water. The aqueous mixture was extracted with 4 × 300 ml of ether. The combined organic extracts were washed with 4 × 300 ml of water and then dried ($MgSO_4$). Concentration of the ethereal solution and short-path distillation gave 2.45 g (19.3%) of a 36:64 ratio of 2-ethyl-2*H*-1-benzopyran (**7**) and 3,4-dihydro-2*H*-1-benzoxocin (**6**), bp 80–90° (0.35 mm). The residue was chromatographed over silica gel. Elution with benzene–hexane (1:1) gave 11.8 g (32.6%) of 4-(*o*-α,α-diethoxymethylphenoxy)butyldiphenylphosphine oxide (**8a**).

The 2-ethyl-2*h*-1-benzopyran (**7**) was shown to be identical (boiling point, ir, nmr) with an authentic sample prepared in these laboratories as previously described.[2]

3,4-Dihydro-2*H*-1-benzoxocin (6). An analytically pure sample (99.5% by glc), obtained from the 127° DMF run (Table I), had bp 77° (0.55 mm); n^{25}D 1.5768; nmr (neat) δ 1.4–1.9 (m, 2, C_3 methylene), 2.0–2.35 (m, 2, C_4 methylene), 3.8 (t, 2, J = 5 Hz), 5.5 (d of triplets, 1, $J_{5,4}$ = 4, $J_{5,6}$ = 11.5 Hz), 5.98 (d of triplets, 1, $J_{6,4}$ = 1, $J_{6,5}$ = 11.5 Hz), 6.5–7.1 (m, 4, C_6H_4).

Anal. Calcd for $C_{11}H_{12}O$: C, 82.46; H, 7.55. Found: C, 82.75; H, 7.57.

4-(*o*-α,α-Diethoxymethylphenoxy)butyldiphenylphosphine Oxide (8a). An analytically pure sample was obtained by crystallization from benzene–hexane: mp 112–113°; nmr ($CDCl_3$) δ 1.15 (t, 6, CH_3), 1.67–2.68 (m, 6, methylenes), 3.52 (q, 2, $-OCH_2CH_3$), 3.50 (q, 2, $-OCH_2CH_3$), 3.90 (t, 2, $-OCH_2-$), 5.69 [s, 1, $-CH(O)(O)$], 6.65–7.95 ppm (m, 14, aromatic); ir (KBr) ν 1190 cm^{-1} (P=O); *m/e* 452.

Anal. Calcd for $C_{27}H_{33}O_4P$: C, 71.65; H, 7.35. Found: C, 72.02; H, 7.12.

4-(*o*-α,α-Dimethoxymethylphenoxy)butyldiphenylphosphine oxide (8b) was obtained similarly from the reaction of **5** with methanolic sodium methoxide and purified by crystallization from benzene–hexane: mp 118.5–120°; nmr ($CDCl_3$) δ 1.80–2.67 (m, 6, methylenes), 3.28 (s, 6, $-OCH_3$), 3.96 (t, 2, $-OCH_2-$), 5.57 [s, 1, $-CH(O)(O)$], 6.70–7.96 ppm (m, 14, aromatic); ir (Nujol) ν 1180 cm^{-1} (P=O); *m/e* 424.

Anal. Calcd for $C_{25}H_{29}O_4P$: C, 70.75; H, 6.84. Found: C, 70.76; H, 6.67.

3,4,5,6-Tetrahydro-2*H*-1-benzoxocin (9). The hydrogenation of 3,4-dihydro-2*H*-1-benzoxocin (**6**) was accomplished quantitatively in 1 hr, in methanol over 10% Pd/C: bp 58–59° (0.6 mm); n^{25}D 1.5321; nmr (neat) δ 1.1–1.7 (m, 6, $C_{3,4,5}$ methylenes), 2.43–2.70 (m, 2, C_6 $-CH_2-$), 3.80 (t, 2, $-OCH_2-$, J = 5 Hz), 6.65–7.1 ppm (m, 4, C_6H_4).

Anal. Calcd for $C_{11}H_{14}O$: C, 81.44; 8.70. Found: C, 81.44; H, 8.68.

2-Ethyl-2*H*-1-chroman (10) was obtained quantitatively from 2-ethyl-2*H*-1-benzopyran (**7**) as described in the previous experiment for the conversion of **6** to **9**. An analytically pure sample had bp 36–42° (0.1 mm); n^{25}D 1.5252 [lit.[3] bp 116° (16 mm); n^{25}D 1.5250]; nmr (neat) δ 0.9 (split t, 3, $-CH_3$, J = 7 Hz), 1.2–1.9 (m, 4, $-CH_2CH_3$ plus C_3 methylene), 2.35–2.7 (m, 2, C_4 methylene), 3.4–3.82 (m, 1, CH), 6.42–7.07 ppm (m, 4, C_6H_4).

Acid Hydrolysis of 4-(*o*-α,α-Diethoxymethylphenoxy)butyldiphenylphosphine Oxide (8a) to 4-(*o*-Formylphenoxy)butyldiphenylphosphine Oxide (15). The aldehyde **15** was obtained quantitatively by hydrolysis (3 hr) with dilute hydrochloric acid in methanol and crystallization by the addition of ether. Recrystallization from benzene–hexane gave a pure sample: mp 113–114°; ir (KBr) ν 1690 (C=O), 1190 cm^{-1} (P=O); nmr ($CDCl_3$) δ 1.50–2.70 (m, 6, $C_{1,2,3}$ methylenes), 4.05 (t, 2, $-OCH_2-$, J = 5.5 Hz), 6.75–8.05 (m, 14, aromatic), 10.4 ppm (s, 1, $-CHO$). Reaction of **8b** in the same manner gave the same results.

Basic Hydrolysis of 5 to Give 15. The salt **5** (0.04 mol) was heated at 90–100° for 3 hr in a solution of 0.05 mol of NaOH in 100 ml of water. The cooled reaction mixture was extracted with 3 × 200 ml of benzene. The combined organic extracts were washed with 3 × 200 ml of water, dried ($CaCl_2$), and concentrated. The residue was crystallized from benzene–hexane to give 5.0 g (33%) of pure **15**, mp 113–113.5°, identical in all respects with the sample obtained in the previous experiment.

4-(*o*-α,α-Diethoxymethylphenoxy)butyltriphenylphosphonium Bromide (16). A mixture of 4-salicyloxybutyltriphenylphosphonium bromide (**5**, 10.5 g, 0.02 ml), triethyl orthoformate (3.2 g, 0.022 mol), 0.05 g of powdered ammonium bromide, and 10 ml of anhydrous ethyl alcohol was heated under reflux for 2.5 hr. Ethanol (5 ml) was removed by distillation and the residue was added dropwise, with vigorous stirring, to 250 ml of anhydrous ether. A quantitative yield of the salt **16** was obtained: mp 178–181°; nmr ($CDCl_3$) δ 1.07 (t, 6, $-CH_3$, J = 7 Hz), 1.60–2.55 (m, 4, $C_{2,3}$ methylene), 3.47 [q, 4, $-CH(OCH_2CH_3)_2$], 4.12 (m, 4, $C_{1,4}$ methylene), 5.67 [s, 1, $-CH(OC_2H_5)_2$], 6.75–8.15 ppm (m, 19, aromatic).

Anal. Calcd for $C_{33}H_{38}O_3PBr$: C, 66.78; H, 6.45; P, 5.22. Found: C, 67.07; H, 6.81; P, 5.34.

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Registry No.—4, 40359-43-1; **5,** 52032-55-0; **6,** 52032-56-1; **7,** 7734-60-3; **8a,** 52032-57-2; **8b,** 52032-58-3; **9,** 51060-43-6; **10,** 7734-61-4; **15,** 52032-59-4; **16,** 52032-60-7; 1,4-dibromolutane, 110-52-1; salicylaldehyde, 92-02-8; sodium ethoxide, 141-52-6; sodium methoxide, 124-41-4.

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